Vol. 3 February, 1960 No. 1

MINERALOGICAL JOURNAL

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The Mineralogical Society of Japan
Chigaku Center, Niban-chô,
Chiyoda-ku, Tokyo

MINERALOGICAL JOURNAL

published by

The Mineralogical Society of Japan

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Printed in Japan

MINERALOGICAL JOURNAL, VOL. 3, NO. 1, PP. 1-8, FEB., 1960

THE X-RAY POWDER PATTERNS AND THE LATTICE CONSTANTS OF NATURAL CRISTOBALITES

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ABSTRACT

Three natural cristobalite specimens from different localities in Japan were prepared by treating the raw specimens with fluoboric acid. The sample obtained from "Beppu Hakudo" gave rather sharp X-ray powder patterns in which many of the lines in the region of back reflection which had first been described in the writer's previous paper were recognized again. The lattice constants of the sample are larger than those of the standard artificial cristobalite by 0.30%. This discrepancy is attributable to the simultaneous crystallization of a small amount of tridymite in the sample. The patterns of the other two natural specimens are rather obscure, but their spacings are in agreement with the standard values within the experimental errors.

Introduction

Quite a few papers have been published on the lattice constants of cristobalite, which are not so accurate as those of quartz owing to the difficulty of obtaining good crystals. As reported in his previous paper[10], the author was able to obtain good crystals of cristobalite by treating some used silica bricks with fluoboric acid and up to eighty seven powder lines including back reflections were clearly observed of them. By treating natural cristobalite rocks in the same way he also obtained good crystals, and in this paper, the results of study on these natural cristobalites will be described.

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Preparation of samples

Three natural cristobalites of different localities in Japan were prepared. They had been treated with fluoboric acid to obtain good samples for precise X-ray experiment.

Sample D was obtained from a specimen of "Beppu Hakudo" which was being shipped from the Beppu hot springs area as a raw material for Portland cement and is considered to be an alteration product of andesite due to acidic hydrothermal solution containing sulphuric acid[4]. The chemical analysis of the specimen revealed that it contained 94.84% SiO_2, a small amount of Al_2O_3, Fe_2O_3 and TiO_2, and a trace of MgO, CaO and SO_3, with 1.76% ignition loss.

Sample E was obtained from a specimen of the "acid earth" which was being shipped from Nakajô, Niigata Prefecture. It is essentially a montmorillonite clay produced from acidic volcanic rocks in the presence of weakly acidic circulating water, and comprises a small amount of quartz.

Sample F was obtained from a siliceous white powder collected at the Ibusuki clay deposit[6] in the vicinity of Ibusuki Spa, Kagoshima Prefecture. It is the mixture of cristobalite, kaolinite, quartz and alunite, and is considered to be an alteration product of andesites due to solfatara gas or acidic hydrothermal solution.

X-ray powder lines and lattice constants

The diffraction angles of X-ray powder lines were measured at the room temperature of 30°±2°C. by using a "Norelco" X-ray diffractometer. Filtered CuK radiation was used with an X-ray tube running at 32 kV., 18 mA. Operation condition of ratemeter was as follows: scale factor 4, multiplier 1, and time constant 8 sec. Divergence and scatter slits were 1° for front reflection and 4° for back reflection, and the receiving slit was 0.006 inches. The

Table 1. Detected powder lines of cristobalites.

*hkl**	A and B				C	D	E	F
	2θ**	Relative intensities	Spacings					
			Observed	Calculated††				
101	22.00†	100	4.04 Å	4.040 Å	+	+	+	+
110?	25.34	2	3.515	3.517				
111	28.45	25	3.134	3.136	+	+	?	+
102	31.44	30	2.843	2.842	+	+	+	+
200	36.12	30	2.485	2.487	+	+	+	+
112	36.41	9	2.466	2.468		+	+	
201	38.47	1	2.340	2.340				
210	40.32	0.2	2.2	2.224				
211	42.70	7	2.118	2.118	+	+	+	+
202	44.88	6	2.020	2.020	+	+	+	+
113	47.08	10	1.930	1.930	+	+	+	+
212	48.62	13	1.872	1.871	+	+	+	+
220	52.02	1	1.758	1.758				
004	52.93	1.5	1.730	1.732				
203	54.23	6	1.691	1.692	+	+		
104	56.3	1	1.634	1.635				
301	57.1	10	1.613	1.612	+	+	+	
213	57.5	3	1.603	1.602				
222, 310	58.8	1	1.570	1.568, 1.573				
311	60.32	5	1.534	1.534	+	+	+	
302	62.09	6	1.495	1.495	+	+	+	
312	65.13	5	1.432	1.432	+	+	+	
204	65.72	2	1.421	1.421				
223	66.91	2	1.398	1.399	+	+		
214	68.77	4	1.365	1.366	+	+	+	
321	69.50	2	1.352	1.353		+		
303	69.90	1	1.346	1.347				
105	70.63	3	1.333	1.334	+	+		
313	72.80	4	1.299	1.300	+	+		
322	74.00	5	1.281	1.281	+	+		
224	77.24	1	1.2341		+	+		
401	77.97	2	1.2243		+	+		
410	79.35	3	1.2064		+	+		
323	81.12	3	1.1846			+	+	
215	81.84	3	1.1759		+	+		
314	82.84	1	1.1643		+	+		
331	83.52	0.8	1.1565					
420	87.81	1	1.1107		+	+		
421, 116	89.22	4	1.0968		+	+	+	+
225	90.1 †	0.2	1.0883	1.0883				
324	91.07	0.4	1.0793	1.0791	+		+	
413	92.1 †	0.2	1.0669	1.0693				

422	93.31	0.9	1.0591	1.0589	+	+		
333	94.91	0.4	1.0455	1.0454	+			
315	95.62	0.5	1.0396	1.0396		+		
404	99.38	0.2	1.0101	1.0101				
423	100.46	0.7	1.0021	1.0021		+		
430	101.46	0.8	0.99493	0.9948				
414	102.18	2	0.98986	0.9899	+	+	+	+
431	102.88	0.8	0.98503	0.9847				
325	104.00	0.3	0.97745	0.9775				
107	105.03	0.9	0.97069	0.9706				
334				0.9708		+		
511	105.7 †	0.8	0.96636	0.9660				
432	107.32	1.2	0.95622	0.9561	+	+		
117	107.9 †	0.1	0.95268	0.9526				
306	108.79	0.4	0.94736	0.9474		+		
424	110.75	0.2	0.93603	0.9358				
316	111.71	0.5	0.93067	0.9307		+		
405	112.64	0.3	0.92561	0.9254		+		
207	113.85	0.3	0.91920	0.9192		+		
433	114.93	1	0.91363	0.9136	+	+	+	
415	115.7 †	0.4	0.90975	0.9098				
217	116.92	1	0.90375	0.9041		+		
513	118.00	0.4	0.89860	0.8986				
522	119.34	0.6	0.89239	0.8924				
326	120.95	0.4	0.88520	0.8854		+		
425	125.30	0.8	0.86719	0.8673				
227	126.58	1	0.86226	0.8624	+	+		
523	127.78†	0.2	0.85778	0.8576				
108	129.16†	0.5	0.85281	0.8530	+	+		
442	129.36	0.8	0.85210	0.8523			+	
531	130.96†	0.2	0.84660	0.8466				
416?	133.84	0.4	0.83726	0.8341	+			
600	136.65†	0.04	0.82885	0.8290				
601	138.76	1	0.82297	0.8230	+	+		
208	140.82	1	0.81757	0.8177	+	+		
610				0.8178				
435	144.93	0.3	0.80778	0.8081		+		
602	145.55	1.5	0.80642	0.8062	+	+		
515	148.34	0.7	0.80061	0.8004		+		
533?	149.36†	0.3	0.79863	0.8002				
612?	150.05	2	0.79732	0.7958	+	+	+	+
621	160.76	1	0.78123	0.7814				
603	161.81†	0.2	0.78005	0.7800				

Cu $K\alpha$, 32 kV., 18 mA. Divergence and scatter slits: 1° for front reflection and 4° for back reflection. Receiving slit, 0.006 inches. Scanning speed 1/8° per minute. Scale factor 4, multiplier 1, and time constant 8 sec.

Samples used had been treated with fluoboric acid[10].

A: Obtained from a silica brick which had been used for three years in the arch of a glass furnace tank[10].

B: Obtained from a silica brick which had been used in the arch of an open-hearth furnace[10].
C: Obtained by heating the red chert from Tamba at 1600° ±50°C. for 45 min. in a reducing atmosphere[10].
E: Obtained from the "Beppu Hakudo" from Beppu, Ôita Pref.
D: Obtained from the acid earth from Nakajô, Niigata Pref.
F: Obtained from the siliceous white powder from the Ibusuki clay deposit in Kagoshima Pref.

* Indices in boldface figures are those which had not been described before the writer's previous work[10].

** Calibrated using Si plate. The corresponding angles of A and B coinsided with each other within the experimental error of 0.02°.

† The experimental errors for those angles were somewhat larger than the above mentioned.

†† On the basis of the following lattice constants (Table 2):
a_0=4.9733 Å., c_0=6.9262 Å.

scanning speed of the G. M. counter was 1/8° per minute.

Detected powder lines of the natural cristobalites are listed in Table 1 together with the data of the standard artificial samples A and B[10]. Many of the lines in the region of back reflection which were first described in the writer's previous paper are recognized again with natural cristobalites especially with Sample D.

Among the natural cristobalites Sample D gives rather sharp reflection lines, and all the detected lines except (612) have been indexed in terms of the tetragonal lattice. The lattice constants are calculated as a_0=4.988Å. and c_0=6.944Å., which are larger than those of the standard artificial cristobalite by 0.30%, while the ratio of c_0 to a_0 is practically identical with that of the standard (Table 2).

The powder patterns of E and F were rather obscure and the experimental error of the Bragg angles for them was ±0.04°. Taking the error into consideration, there is no discrepancy in spacings between these natural cristobalites and the standard (Table 2).

Table 2. Lattice constants of natural and artificial cristobalites.

Observer	Temp. at	a_0	c_0	$c_0 : a_0$	Sample
Nieuwenkamp[7]	—	4.97 Å	6.93 Å	1.39	
Jay[3]	22°C	4.9715	6.9193	1.392	Cristobalite from a used silica brick.
Swanson & Tatge[8]	27	4.973	6.95	1.398	Amorphous silica powder heated 2 hr. at 1420°C.
Tokuda[10]	30	4.9733 ±0.0003	6.9262 ±0.0018	1.3926	A and B*.
Tokuda	30	4.988	6.944	1.392	D*.
	30	4.97	6.93	1.39	E*.
	30	4.98	6.93	1.39	F*.

* Refer to the footnote of Table 1.

Considerations on the discrepancy in the lattice constants of cristobalite

Table 2 shows the lattice constants of cristobalite hitherto reported. Taking the thermal expansion of cristobalite into consideration, the result obtained by the writer with the standard artificial samples A and B are in good agreement with those obtained by Jay[3].

The recent investigation by Flörke[1] implies that the cristobalite which has been crystallized simultaneously with tridymite may have more lattice defects hence larger spacings. In this respect, it is worthy of notice that several small peaks due to tridymite-S[2] were detected in the neighbourhood of the (101) peak of cristobalite in the X-ray powder patterns of Sample D (Fig. 1). The discrepancy in the spacings between Sample D and the standard cristobalite may be attributed to the simultaneous crystallization of a small amount of tridymite in the "Beppu Hakudo".

The cause of rather large values reported by Swanson and Tatge[8] may be interpreted in the same way, because they recognized some reflections from tridymite in the patterns of the cristo-

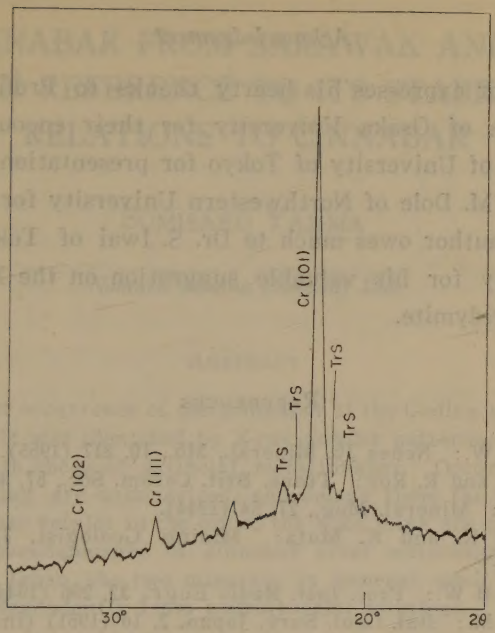

Fig. 1. X-ray powder patterns of Sample D, obtained from the "Beppu Hakudo".
Cr: cristobalite, TrS: tridymite-S[2].

balite. But in this case, another factor should be taken into consideration, that is, the temperature of formation of cristobalite. According to the writer's experiment[9], the lower the temperature of formation, the larger the lattice constants of the produced cristobalite, and the temperature of 1420°C. at which their cristobalite was formed seems too low to produce a standard sample.

The writer is inclined to draw the following conclusion from the above considerations:

The standard lattice constants of cristobalite are represented by those obtained by Jay as well as by the writer. The lattice constants of natural cristobalites are the same as the standard unless they have been crystallized simultaneously with tridymite, in which case the constants may have larger values.

Acknowledgement

The author expresses his hearty thanks to Prof. T. Ao and Prof. I. Nitta of Osaka University for their encouragement, to Prof. S. Iwao of University of Tokyo for presentation of samples, and to Prof. M. Dole of Northwestern University for his kind revision. The author owes much to Dr. S. Iwai of Tokyo Institute of Technology for his valuable suggestion on the X-ray powder patterns of tridymite.

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Manuscript received September 14, 1959.

Mineralogical Journal, Vol. 3, No. 1, pp. 9–18, Feb., 1960

METACINNABAR FROM SARAWAK AND JAPAN, WITH REFERENCE TO ITS STABILITY RELATIONS TO CINNABAR

Sumisaku Yajima

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Abstract

The mode of occurrence of metacinnabar at the Gading mine, Sarawak, is described. It was identified by X-ray powder patterns which were in good accord with those of artificial metacinnabar. Occurrences of the mineral in Japan are summarized, and among them the oolitic texture and metacinnabar veinlet in the ore of the Nikô mine are noticeable. Although some pseudomorphs of cinnabar after metacinnabar have been described from Japan, the two minerals, in general, seem to have been precipitated independently from hydrothermal solutions.

Introduction

The so-called black mercury sulphide has been reported to occur with cinnabar, but has seldom been studied in detail. In 1958, the writer had an opportunity to visit the mercury ore deposits of Gading, Sarawak, and collected a specimen containing metacinnabar, which will be described in this paper.

Recently, Frank W. Dickson and George Tunell[2] published their paper concerning the stability relations of cinnabar and metacinnabar. The modes of occurrence of these two minerals in the Gading ores as well as in the ores of Japan imply that they do not always coincide with the relations reported by Dickson and Tunell nor with Lindgren's opinion[5]. Considerations upon these points will also be presented.

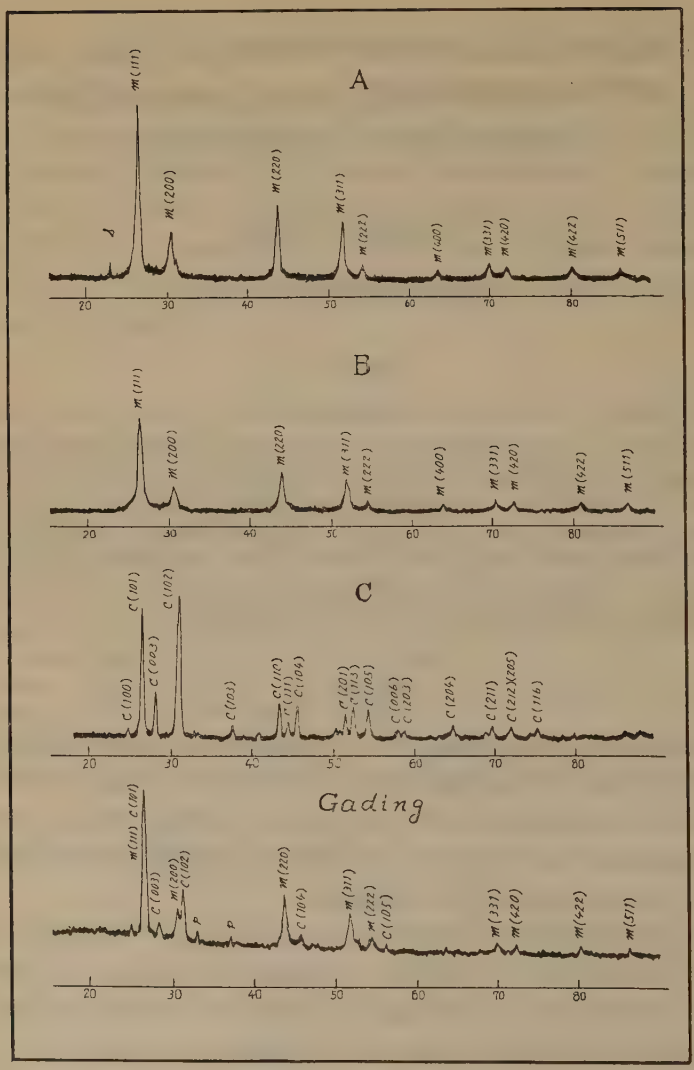

Fig. 1. X-ray powder patterns of mercury sulphides.
A: Artificial metacinnabar, $Hg+S \rightarrow HgS$.
B: Ditto, $HgCl_2+H_2S \rightarrow HgS+2HCl$.
C: Artificial cinnabar.
Gading: Specimen from the Gading mine, Sarawak.
m: Metacinnabar, c: Cinnabar, s: Sulphur, p: Pyrite.

Preparation and X-ray powder patterns of standard samples

Two groups of artificial metacinnabar and one group of artificial cinnabar were prepared as standard samples for X-ray examination.

Sample A is a black powdery mercury sulphide which was obtained by blending the mixture of sulphur powder and mercury at room temperature.

Sample B was prepared by drying the black gelatinous precipitate produced by the reaction between mercuric chloride solution and hydrogen sulphide below 100°C.

These two samples give the X-ray powder patterns of metacinnabar as illustrated in Fig. 1. The patterns were recorded by a "Norelco" X-ray diffractometer.

When the artificial metacinnabar was heated with sodium hydroxide solution on a water bath, colour is gradually changed to scarlet of various tint through dark purplish red. Sample C, the scarlet product, gives the X-ray powder pattern of cinnabar as illustrated in Fig. 1.

The patterns of A, B and C are in good accord with previous works, and imply that metacinnabar will be unambiguously distinguished from cinnabar by means of X-ray powder method.

Mercury deposits of Gading, Sarawak

Gading mercury deposits are situated near the boundary between Sarawak and Kalimantan of Indonesia and about thirty kilometres southwest of Kuching, the capital of Sarawak Kingdom (Fig. 2). Of them, Tegora deposit was first discovered and proved to be of economical value in 1867 by L. V. Helm, the first manager of Borneo Company. The Gading deposits were exploited in the years from 1868 to about 1898. During Japanese occupation from 1942 to 1945, both alluvial and eluvial deposits of Gading were extensively worked along with some shallow veins at Gading. In March, 1958 when

Fig. 2. Locality of the Gading mine, Sarawak.

the writer visited the deposits, the mining area had been covered with jungle, and only piles of waste, a broken adit and rusty remains of machines reminded him of the past. Accordingly it was difficult for him to carry on detailed survey over the entire deposits, but he obtained some interesting ore specimens, one of which has been revealed to comprise metacinnabar.

The area is made up of the alternation of black shale and hard sandstone probably of Cretaceous, and of such igneous rocks as micro-granodiorite and dacite. They say that the Cretaceous formation is intruded by those igneous bodies, but the writer could not recognize any field evidence to verify it.

The mercury deposits of Gading are epithermal veins filling brecciated zones in the Cretaceous formation, and dipping 70°～80° east. In many cases the brecciated zone contains fragments of black shales, and where it traverses hard sandstone, the zone adjacent to the vein is silicified and impregnated with cinnabar.

The principal ore mineral in the deposit is cinnabar, which is sporadically accompanied by a small amount of native mercury and

marcasite. The characteristic minerals which distinguish the Gading ore from other mercury ores are realgar and orpiment. Chemical analyses have revealed that most of the Gading ores contain as much arsenic as mercury. Under the microscope, realgar is sometimes intimately associated with cinnabar, and seems to have been precipitated just after the crystallization of quartz and iron sulphide.

Gangue minerals in the Gading deposits are opaline silica and kaolin which is composed of abundant dickite and a small amount of kaolinite as revealed by d.t.a. and X-ray powder patterns.

Metacinnabar from Gading, Sarawak

On a specimen of the breccia ore of Gading, the writer found small cavities, 0.3~0.6 cm. in diameter. They are lined with black mineral aggregates which are apparently separated from cinnabar disseminated over the ore (Fig. 3). Most of the aggregates are of acicular form and composed of fine crystals with submetallic lustre.

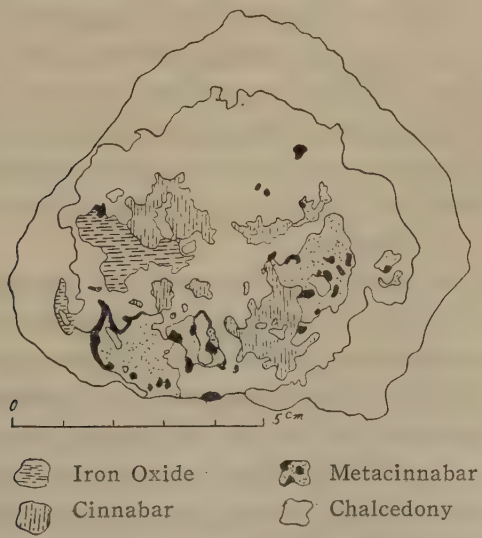

Fig. 3. Metacinnabar-bearing ore specimen from the Gading mine, Sarawak.

The hardness of the black mineral is low and the streak is black. Qualitative chemical analysis revealed the presence of mercury and sulphur in the mineral.

The X-ray powder patterns of the mineral aggregate are compared with those of artificial metacinnabar and cinnabar in Fig. 1. It is evident that the aggregate is essentially composed of metacinnabar accompanied by a small amount of cinnabar.

The aggregate of metacinnabar in the Gading ore is distinguished from those of cinnabar in the same ore by its acicular form and mode of occurrence, and it implies that the metacinnabar has been crystallized independently of the cinnabar, but not as the pseudomorph after cinnabar. It also seems improbable that the cinnabar has been produced by inversion from the metacinnabar.

Occurrences of metacinnabar in Japan

Most of the so-called black mercury sulphide minerals accompanying cinnabar ores in Japan have been revealed to be cinnabar. Kinoshita[4], and Takabatake and Hashimoto[8] reported that many black mercury sulphide minerals turned red in colour when scratched, and under the microscope, transition from black to transparent red mineral was usually observed. Umegaki[9] pointed out that it is difficult to distinguish metacinnabar from cinnabar by means of microscopic observation only.

Recently, Ushizawa[10] reported that most of the black mercury sulphide minerals which looked like metacinnabar were identified as cinnabar through observation with reflecting light.

Some remarkable pseudomorphs of cinnabar after metacinnabar have been described by Sakurai and Sorita[7] from the Niû mine, Mié Prefecture, and the Chihaya mine, Osaka Prefecture (Fig. 4). The pseudomorph from Niû is cubic in crystal habit, while that from Chihaya is octahedral with both positive and negative tetrahedra, attaining to 1～2 mm. in diameter. They were once described

as metacinnabar[3], but subsequent examination of X-ray powder photographs and streak tests revealed them to be cinnabar[7].

On the other hand, metacinnabar has been identified in the ores from the Yamato mine[8], Nara Prefecture (Fig. 4), the Nakasé mine[7], Hyogo Prefecture (Fig. 4) and Okéto and Nikô mines[12] in Hokkaido (Fig. 5). Among them those from the last two mines contain considerable amount of metacinnabar, and have been studied by the writer.

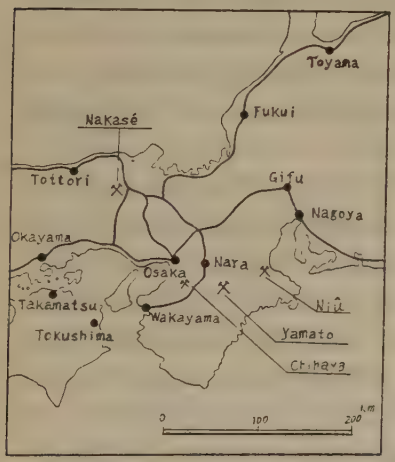

Fig. 4. Locality map for the Nakasé, Chihaya, Yamato and Niû mines.

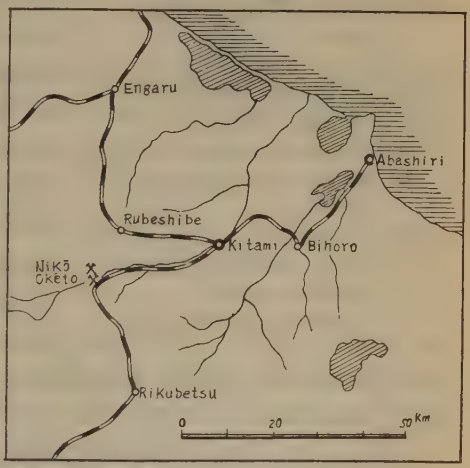

Fig. 5. Locality map for the Niko and Okéto mines.

Fig. 6 shows the mode of occurrence of metacinnabar in an ore specimen from the Nikô mine. The ore is mainly composed of alunite impregnated with metacinnabar and cinnabar, and sporadically traversed by metacinnabar or calcite veinlet. The impregnated metacinnabar forms the crust of oolite several milimetres across and the crust is enveloped by cinnabar grains.

This mode of occurrence suggests the similar relation between the crystallizations of the two mercury sulphides as has been implied by the Gading ore of Sarawak, that is, the metacinnabar from

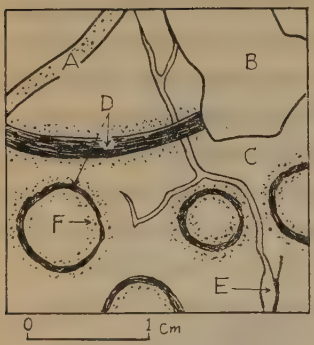

Fig. 6. Texture of the metacinnabar-bearing ore of the Nikô mine, Hokkaido.
A: Quartz veinlet,
B: Breccia of silicified liparite,
C: Alunite,
D: Metacinnabar,
E: Calcite veinlet,
F: Cinnabar impregnation.

the Nikô mine may have been crystallized independently of cinnabar.

In the Okéto mine there is a small liparite dike impregnated with mercury sulphides. Metacinnabar predominates in the central part of the dike, while toward the periphery the ratio of cinnabar to metacinnabar increases. The metacinnabar forms small oolite several milimetres across and the outer zone of the oolite gradually turns to cinnabar.

According to Sakurai and Sorita[7], the metacinnabar from the Nakasé mine is similar in occurrence to that of Okéto. It forms the nucleus of small grains which impregnate berthierite-bearing gold-quartz vein and is enveloped by dark red cinnabar.

The metacinnabar of the Yamato mine was found by Takabatake and Hashimoto[8] in a banded ore at the Seventh level, forming distinct bands separated from the cinnabar bands.

Consideration of stability relations between metacinnabar and cinnabar

As early as 1912, Allen, Crenshaw and Merwin[1] studied the stability relations between metacinnabar and cinnabar and concluded that the former was not stable at temperatures up to 570°C. Rinse[6] measured the dissociation pressures of mercury sulphides at temperatures ranging from 331°C. to 651°C., and concluded that cinnabar inverts to metacinnabar at 386°±2°C.

Recently Dickson and Tunell[2] performed an exact measurement of the inversion with the result that the inversion point between metacinnabar and cinnabar is 344°C. at one atmospheric pressure, and that the presence of iron or zinc in metacinnabar, which is a rather common case with natural metacinnabar, lowers the inversion temperature. They also noted that natural metacinnabars in general were deposited earlier than cinnabar and in rare instances the two minerals were crystallized from solutions simultaneously or in alternation, but that metacinnabar cannot be considered as an alteration product of cinnabar.

The pseudomorphs of cinnabar after metacinnabar which have been found at the Niû and Chihaya mines may be considered as remarkable evidence in nature verifying the experimental results by Dickson and Tunell. The oolitic metacinnabar from the Okéto mine and the metacinnabar grains from the Nakasé mine, both of them being enveloped by cinnabar, may be interpreted according to their results as well.

On the other hand, the mode of occurrence of metacinnabar in the ores of the Gading, Nikô and Yamato mines, lacking evidence for inversion between metacinnabar and cinnabar, implies that the two minerals crystallized independently.

These ores are of epithermal origin, and the metacinnabar must have been precipitated from hydrothermal solutions at rather low temperatures. As is evident from the experiment as well as from the natural occurrence in the deposits from hot springs at Amedee[2], California and Boiling Springs[2] [11], Idaho, metacinnabar may be easily deposited from solutions at lower temperatures. It is highly probable that fluctuations in the pH-value of the hydrothermal solution made the change of phase of the crystallizing mercury sulphide during the mineralization of the Gading, Nikô and Yamoto deposits.

Since most of the mercury ore deposits are considered to be of epithermal origin, it is desirable to study the stability relations between metacinnabar and cinnabar in the presence of various solu-

tions at temperatures corresponding to epithermal conditions.

In conclusion, the writer wishes to thank Mr. E. Ishii and T. Mariko for their assistance in X-ray experiments, and Prof. Z. Harada and Prof. N. Katayama for their revision of this report.

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Manuscript received November 30, 1959.

Mineralogical Journal, Vol. 3, No. 1, pp. 19-29, Feb., 1960

DIELECTRIC PHENOMENA OF SMOKY QUARTZ, NATURAL AND X- AND γ-RAY IRRADIATED

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Abstract

Dielectric dispersion of natural smoky quartz was observed in the audio-frequency region at about 400°C. The enthalpy of activation for the dielectric relaxation was about 50 kcal/mole in contrast with a value of about 20 kcal/mole for natural colourless quartz. By thermal bleaching, the enthalpy of activation falls to the value for natural colourless quartz.

Exposed to X-rays, the bleached quartz coloured again and the value of activation enthalpy recovered, and to γ-rays of about 10^5 to 10^7 r. in total dose, coloration also attained, but the effect was different from that by X-rays. A linear relation was found between the energies of activation and the logarithms of the total doses of irradiation.

Probable molecular mechanisms responsible to these dielectric phenomena were proposed. The rôle of water molecules trapped in the structure was discussed.

Introduction

In the preceding paper[1], dielectric dispersion at above room temperature for colourless and transparent natural quartz was discussed from a standpoint of a rôle of water molecules and ions included in some lattice defects as impurities. We have now studied the dielectric dispersion phenomena of smoky quartz, natural and irradiated, at higher temperatures to confirm our previous conclusion on the mechanism of dielectric relaxation. The results on smoky quartz were distinctly different from those on colourless one as had been expected. The present paper is a brief report on these experimental results.

Experimental method

The apparatus used was essentially the same as that previously reported[2]. Dielectric dispersion was measured mainly in the audio-frequency region, namely between 0.3 and 10 kc., using a set of capacity-resistance bridge and C-R oscillator. In the radio-frequency region, a Q-meter was used. A crystal plate, about 1.5 mm. thick, cut perpendicular to the c-axis was inserted into a platinum-foiled condenser without electrode-pasting.

Specimens and conditions of irradiation

Crystals of natural smoky quartz from Naégi, Gifu Prefecture, and from Ishikawa, Fukushima Prefecture, Japan were used for the present experiment.

As thermal bleaching occurs above about 300°C., colourless specimens for X-ray or γ-ray irradiation were prepared by heating at 500°C. for several hours.

The conditions of X-ray irradiation were as follows: (1) Unfiltered CuK radiation at 32 kV., 18 mA. with the dose rate of 75 r/min.; the total dose was about 10^5 r. (2) White radiation from tungsten at 45 kV., 40 mA. with the dose rate of 240 r/min.; the total dose was also 10^5 r. The temperature of the specimen during irradiation increased considerably, especially in the latter case, up to about 100°C.

The conditions of γ-ray irradiation were as follows; 1540 curies of the Co^{60} source was used. The specimen sealed in a small glass vessel was immersed into a water pool wherein the radiation source had been placed at the bottom and was irradiated for a necessary period of time. Therefore, no temperature elevation took place in this case. The total doses ranged from 5.1×10^5 to 1.5×10^7 r. with the dose rate of 1.1×10^4 to 1.5×10^5 r/hour.

Results

(1) *Natural smoky quartz.* A representative result of the dielectric dispersion along the c-axis for natural smoky quartz is shown in Fig. 1, and clearly indicates that the temperature at which the dispersion appears for smoky quartz is much higher than that for colourless one. The activation energies and entropy calculated from the observed data according to Eyring's relation for dielectric relaxation are listed in Table 1. The activation enthalpy, ΔH^*, for smoky quartz was also much higher than that of colourless one. However, the bleached specimen gave a small value of activation enthalpy, which was nearly equal to that of natural colourless one, as shown in Table 1. The dispersion region of bleached but originally smoky quartz is also found in the same

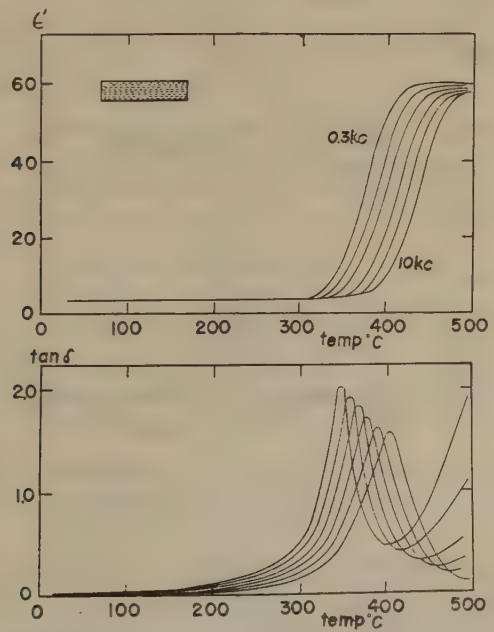

Fig. 1. Dielectric dispersion of a natural smoky quartz at 0.3, 0.5, 1.0, 3.0, 5.0 and 10.0 kc.

Table 1. Activation energies and entropy for dielectric relaxation of natural quartz.

Specimen	ΔH^* (kcal/mole)	ΔF^* (kcal/mole)	ΔS^* (E. U.)
Smoky (Naégi, No. 1)	59.0	24.6	50 at 685°K
Smoky (Naégi, No. 2)	38.0	24.9	20 673
Smoky (Ishikawa)	55.9	24.5	47 663
Bleached (Naégi, No. 1)	26.3	20.9	10 568
Colourless (Brazil)	21.3	18.4	6 503

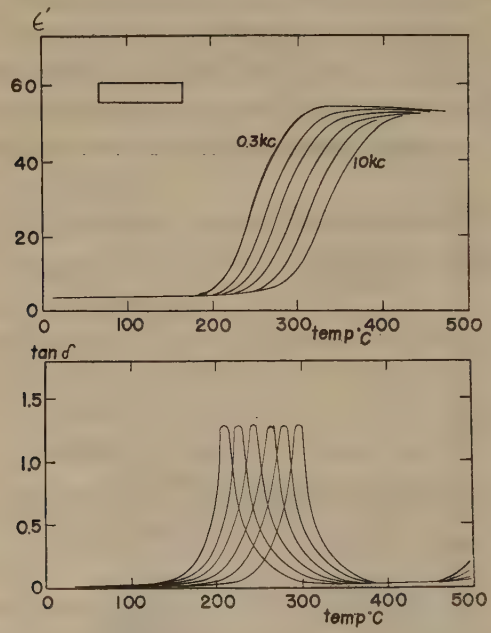

Fig. 2. Dielectric dispersion of a bleached quartz at 0.3, 0.5, 1.0, 3.0, 5.0 and 10.0 kc.

temperature region as that of natural colourless one. The feature of the dielectric dispersion is shown in Fig. 2.

(2) *Coloured smoky quartz caused by X-ray irradiation.* Homogeneous coloration could not be attained in the bleached specimen

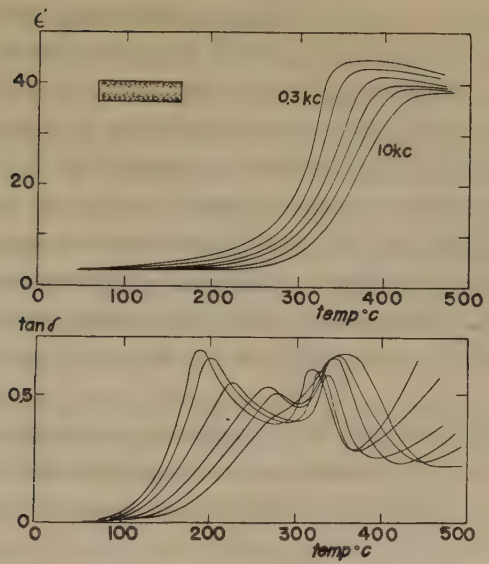

Fig. 3. Dielectric dispersion of an artificially smoked quartz with X-rays of CuK radiation at 0.3, 0.5, 1.0, 3.0, 5.0 and 10.0 kc.

Table 2. Activation energies and entropy for dielectric relaxation of irradiated quartz which was originally smoky and bleached at 500°C.

Radiation	Condition of irradiation		ΔH^* ΔF^*	ΔS^* (at °K)
	dose rate	total dose	(kcal/mole)	(E. U.)
X-rays, Cu K, 32 kV., 18 mA.	75 r/min.	10^5 r.	58.5 23.1 17.2 22.2	58 (613) −10 (523)
X-rays, W, 45 kV., 40 mA.	240 r/min.	10^5	45.0 23.3	35 (614)
γ-rays (No. 1)	1.1×10^4r/h.	5.1×10^5	26.4 21.4	9 (563)
	6.0×10^4	1.7×10^6	32.9 22.0	19 (583)
	3.1×10^4	2.2×10^6	34.4 22.7	19 (593)
	3.1×10^4	5.9×10^6	40.1 23.2	28 (613)
(No. 3)	6.0×10^4	4.0×10^6	32.2 23.0	16 (583)
	1.5×10^5	1.5×10^7	44.4 24.1	33 (613)

by CuK radiation owing to the poor penetrating power of the X-rays into the crystal. In spite of irradiation from both sides, a colourless zone remained in the middle part of the specimen as shown schematically at the corner of Fig. 3. In this case, two kinds of dispersion of tan δ were observed (Fig. 3); one found in the lower temperature region obviously due to the colourless zone in the middle part and the other in the higher temperature region due to the smoky surface-zones. Each of the values of activation energy listed in Table 2 is in a fair agreement with the value for natural colourless quartz and that for natural smoky one, respectively.

With white radiation from tungsten, homogeneous coloration was attained, the lower temperature dispersion just described above being scarecely observed. The feature of dispersion curves is quite

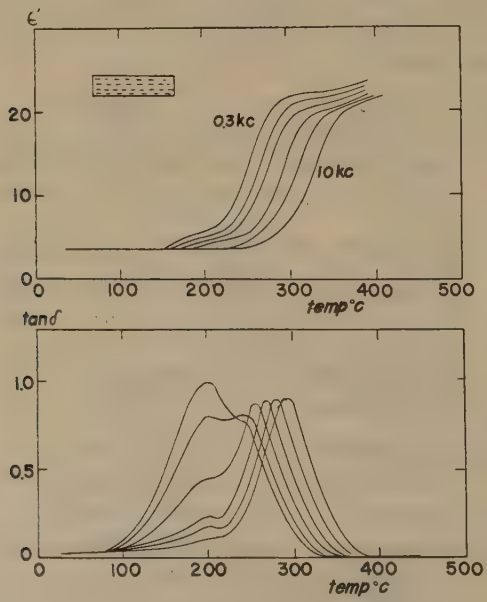

Fig. 4. Dielectric dispersion of a moderately γ-ray irradiated (5×10^5 r.) quartz at 0.3, 0.5, 1.0, 3.0, 5.0 and 10.0 kc.

similar to that of natural smoky quartz.

(3) *γ-ray irradiated quartz.* Homogeneously coloured, smoky quartz was obtained by γ-rays. The dielectric dispersion of a moderately exposed specimen (5.1×10^5 r.) is shown in Fig. 4. There are also two kinds of dispersion of dielectric loss, but the feature of the curves is considerably different from that of X-ray irradiated one. On the low temperature side, a distinctly frequency-dependent but temperature-independent dispersion was found at about 200°C. This characteristic dielectric loss may be attributed to some kind of conduction, presumably conduction of protons liberated from the water molecules by the moderate γ-ray irradiation. The feature of dispersion observed on the high temperature side resembles that of natural smoky quartz.

Specimens strongly exposed with γ-rays of about 6×10^6 r. gave single dispersion in the higher temperature region as shown in

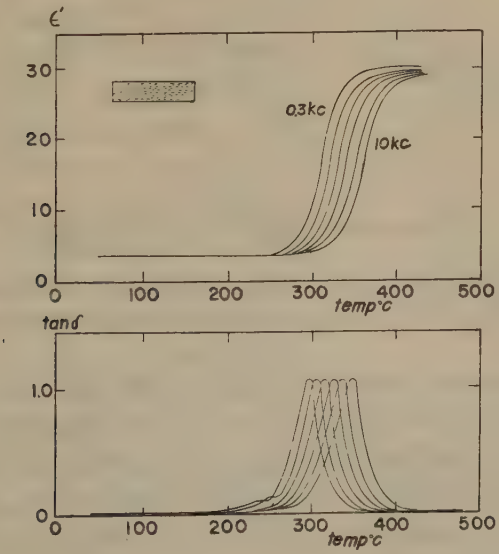

Fig. 5. Dielectric dispersion of a strongly γ-ray irradiated (6×10^6 r.) quartz at 0.3, 0.5, 1.0, 3.0, 5.0 and 10.0 kc.

Fig. 5. This phenomenon is apparently similar to that found for natural smoky quartz.

Thermodynamical data of activation for the specimens irradiated under various conditions are listed in Table 2. It is remarkable that the stronger the total dose of γ-irradiation is, the larger is the energy of activation for dielectric relaxation in the higher temperature region.

Approximately linear relations between energies of activation, or entropies of activation, and the logarithms of total doses were found in our experimental range of γ-irradiation (Fig. 6). One

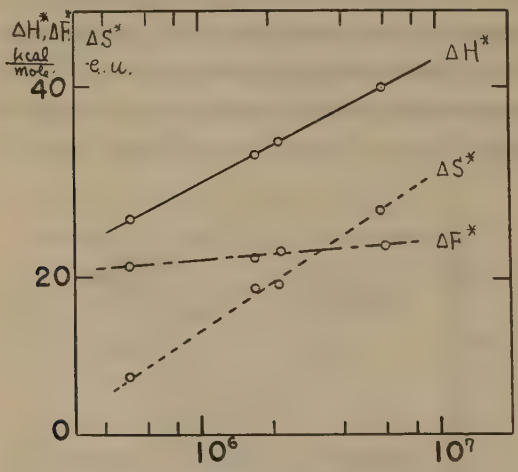

Fig. 6. Linear relations between energies of activation, or entropy of activation, and logarithms of total doses.

specimen originally smoky in colour was used throughout the measurements and bleaching by heating and colouring by irradiation were repeated alternately. Another specimen was examined to confirm the result and such a linear relation was also obtained although the slopes of the lines were not identical with those for the former.

Discussion

In the case of X-ray irradiation, the linear relation between the activation energies and the logarithms of the doses has not been found. Moreover, the enthalpy of activation for dielectric relaxation at higher temperatures has a fixed value irrespective of the kinds of X-ray sources and is larger than that for any one of the γ-ray irradiated specimens.

These facts suggest that the effect caused in the crystal lattice by X-rays is considerably different from that by γ-rays. Then, two different molecular mechanisms should be proposed.

The most appropriate explanation for the linear relation may be to postulate some sort of lattice distortion. Although no evidence of lattice distortion in γ-ray irradiated quartz has yet been reported[3], the radiation energy of γ-rays from the Co^{60} source is much higher than the threshold value of radiation energy for producing atomic displacements[4]. It is reasonable, therefore, that the more intense the dose of irradiation is the more easily are displaced the atoms in the structure, thus rendering the diffusion of impurity ions parallel to the c-axis which is the direction of the spiral axis, more and more difficult as shown schematically in Fig. 7. Consequently, the energy of activation for the ionic displacement or drift to the direction of the applied electric field should be increased gradually with deformation of the trapping cage in the three-diemensional network.

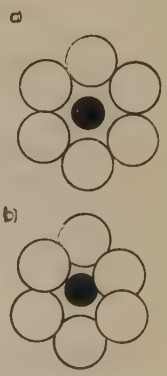

Fig. 7. Schematic model of lattice distorsion projected on the basal plane. The black circle indicates the impurity ion enclosed in the tunnel of quartz lattice. The large open circle indicates oxygen atom of Si-O framework.

Once heated up to 500°C. anneal-

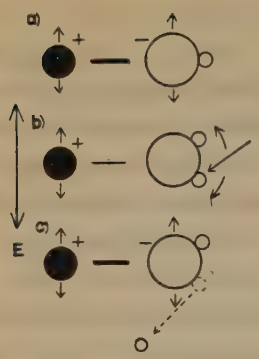

Fig. 8. Schematic models of the interaction of the impurities in quartz lattice.

a) In smoky quartz, Na^+ and OH^- are mutually interact with ion-ion interaction.

b) By heating, recombination of OH^- and H^+ occurs and OH_2 molecule is formed.

c) By absorbing X-rays, strictly speaking by the recoiled electron, OH_2 molecule dissociates to OH^- and H^+. The ion-ion interaction recovers.

ing takes place, so that the value of activation energy recovers.

The effect of X-ray irradiation is quite different from that of γ-ray irradiation mentioned above. The water molecule trapped in some lattice defects may be dissociated by X-ray to H^+ and OH^-. This proton, H^+, has a relatively low kinetic energy and will be readily captured by a neighbouring oxygen atom in the three-dimensional Si-O framework, forming a metastable Si-O-H bond. The OH^- ion will interact with an alkali ion, e. g. Na^+, included nearby in a lattice defect as impurity (see Fig. 8). The ion-ion interaction between Na^+ and OH^- is much larger than the ion-dipole interaction between Na^+ and an OH_2 molecule in colourless quartz. Thus the energy of activation jumps suddenly by X-ray irradiation from a value of 20 kcal/mole up to about 50 kcal/mole. The effect of heating should give the chance of recombination of the free OH^- with H^+ which has been unstably combined with an oxygen atom. Therefore, the recovery of the dielectric phenomena by heating takes the course observed in our experiment.

It is believed that natural smoky quartz usually found in pegmatite has been coloured by natural γ-rays during a long geological period below the bleaching temperature. However, the γ-rays must have been softened in a marked degree by scattering and absorption in the surrounding rock-forming minerals before

the rays reached into the crystal surface. Accordingly, the dielectric phenomena for natural smoky quartz may be similar to those for the specimen artificially smoked by X-rays rather than by γ-rays.

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Manuscript received Sept. 28, 1959.

Mineralogical Journal, Vol. 3, No. 1, pp. 30-41, Feb., 1960

THE MINERALS OF THE NODA-TAMAGAWA MINE, IWATÉ PREFECTURE, JAPAN

II. Pyrochroite Ore (Kimiman-kô) and Its Origin

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Abstract

The manganese ore, locally called "Kimiman" or "Kibiman" by the miners at the Noda-Tamagawa mine, is composed essentially of pyrochroite [$Mn(OH)_2$] hitherto known as a rare mineral. More than 50,000 tons of the pyrochroite ore have been shipped from this mine for metallurgical uses since 1950. The ore consists mainly of fibrous pyrochroite pseudomorph after manganosite with small amounts of manganosite, galaxite, tephroite, rhodochrosite, barite and alabandite. The pyrochroite occurs in scaly aggregates. When fresh, it is white but when exposed in air its colour changes into brown to black. Extinction is parallel to the elongated fiber. Character of zone is positive, $\varepsilon=1.683$, $\omega=1.725$ and $\omega-\varepsilon=0.042$. The unit cell dimensions obtained from the powder data: $a_0=3.323$Å. and $c_0=4.738$ Å. The pyrochroite ore is considered to be hydrothermal alteration product from the manganosite ore, which may have been formed as dissociation product from the rhodochrosite ore of sedimentary type during the period of contact metamorphism of granitic intrusion.

Introduction

While pyrochroite has usually been known as a rare hydrothermal mineral, it occurs in large quantities as a principal manganese ore mineral in the Misago ore body at the Noda-Tamagawa mine.

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Similar occurrences of pyrochroite have often been found in manganese deposits lying in contact aureoles of granitic masses in Japan. In this paper, the mode of occurrence and mineralogical features of pyrochroite ore will be described and further its origin will be discussed.

Occurrence and paragenesis

Misago ore body is the largest manganese ore body ever found in the Noda-Tamagawa mine, and is situated in the central part of the main ore horizon. Since 1950, its lower extension has been systematically exploited along the axes of complicated folds of the ore-bed. The folded ore body plunges into SSW. direction at 35 degrees.

Before the discovery of pure white pyrochroite ores on the lower levels, the brown coloured peculier manganese ores had been mined on the upper level and they were named "Kimiman" or "Kibiman" because its colour resembled that of corn-bearing boiled rice. This Kimiman ore usually occupies the central part of the ore body and is surrounded by narrow zone consisting of tephroite and rhodonite as shown in Fig. 1. The layered structure of the ore body is generally conformable with that of the country rocks.

On the stope face in the old working places the pyrochroite ore looks usually dark brown or black, owing to the surface oxidation. However, immediately after blasting, it becomes light brown or white in colour and laminated or layered structure due to the original bedding of the manganese ore is usually well observed on the stope surface.

In 1953 when the central thicker part of the Misago ore body was stoped on the lower 30 m. level and 45 m. level, the first author could observe a beautiful folded structure of the ore body as shown in Fig. 1. Here the local geologic structure is very complex and both the ore body and country rocks have been sharply folded as shown in the figure. The plunge of the minor foldings

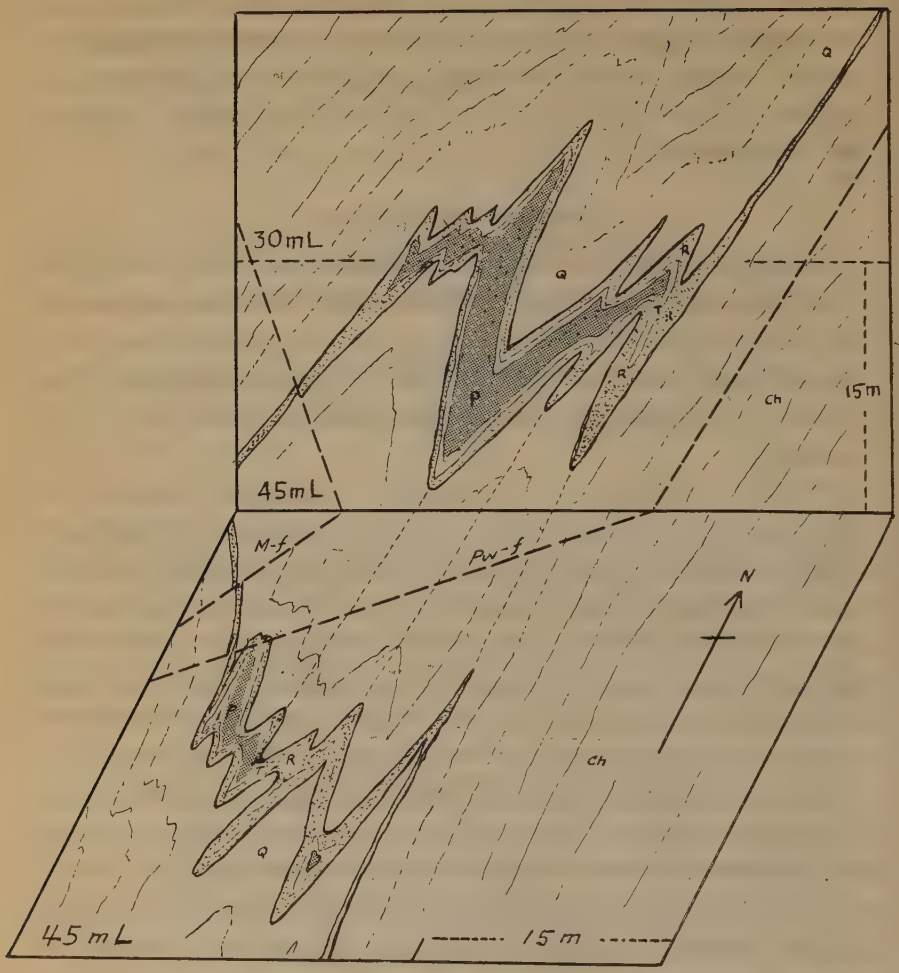

Fig. 1. Diagrammatic horizontal and vertical sections of the Misago ore body, Noda-Tamagawa, showing the folded structure of the pyrochroite ore body and the occurrence of zoned skarn around it.

P: pyrochroite ore, T: tephroite zone, R: rhodonite zone,
Q: massive quartzite, ch: thin-bedded quartzite,
M-f: Misago fault, Pw-f: "Penwithite"-fault.

(a)

(b)

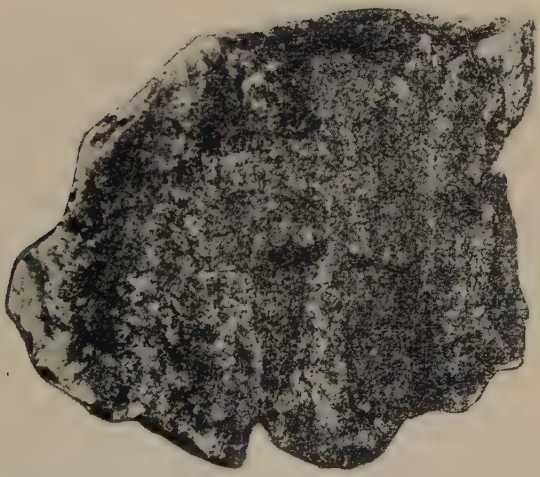

Fig. 2. Macrophotograph of pyrochroite ore (Kimiman-kô) from the Misago ore body, Noda-Tamagawa. (×1)

(a) Unoxidized broken surface of a specimen of the pyrochroite ore. Fresh surface is white in colour.

(b) Oxidized surface of the same specimen. This photograph was taken about one month after breaking of the specimen.

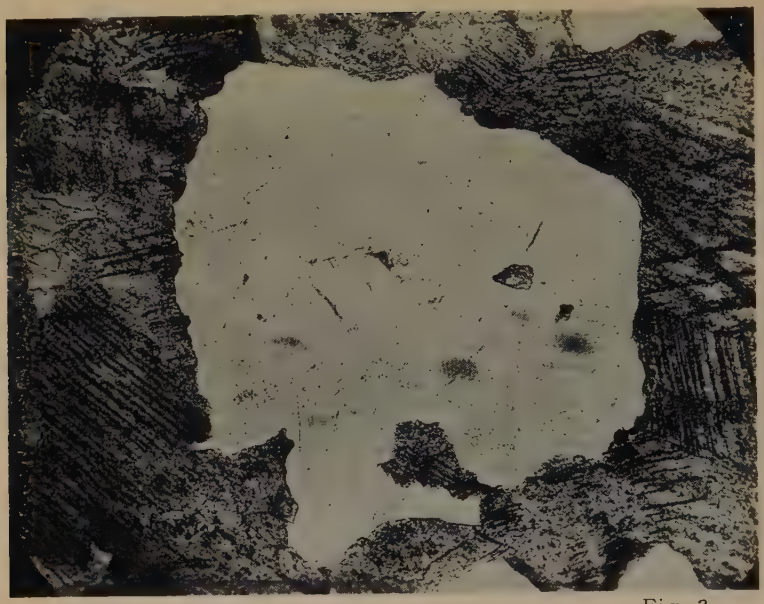

Fig. 3.

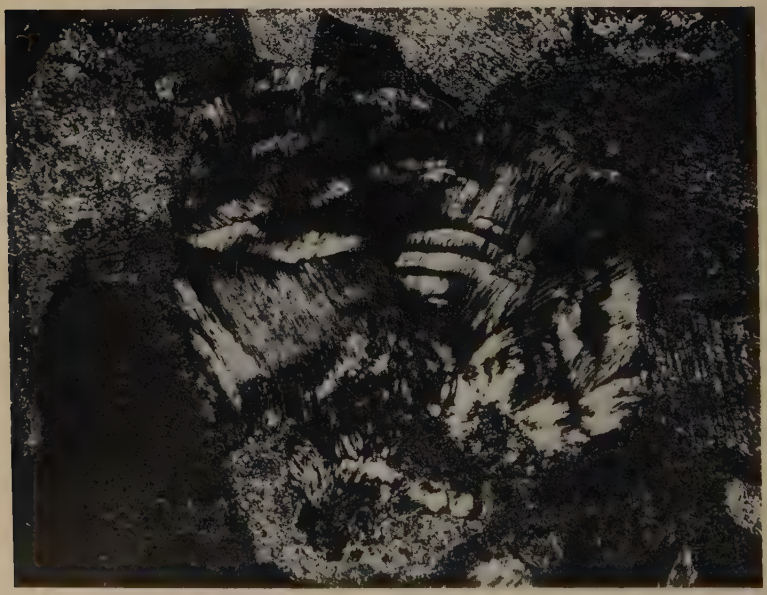

Fig. 4.

Figs. 3 and 4. (×10) Photomicrographs of brucite-marble from the Tul Mi Chung mine, Suan, Korea. Central white part in Fig. 3 is brucite-pseudomorph after periclase. Its surrounding area is composed of calcite. The scaly fibrous aggregate of brucite shown in fig. 4 resembles that of pyrochroite shown in Plate III, Fig. 6. (Fig. 3. Polarizer only. Fig. 4. Crossed nicols.)

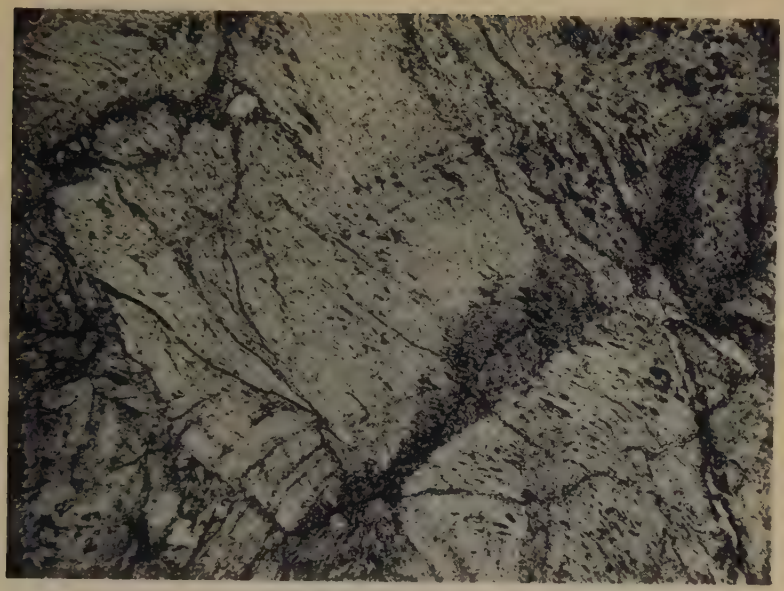

Fig. 5.

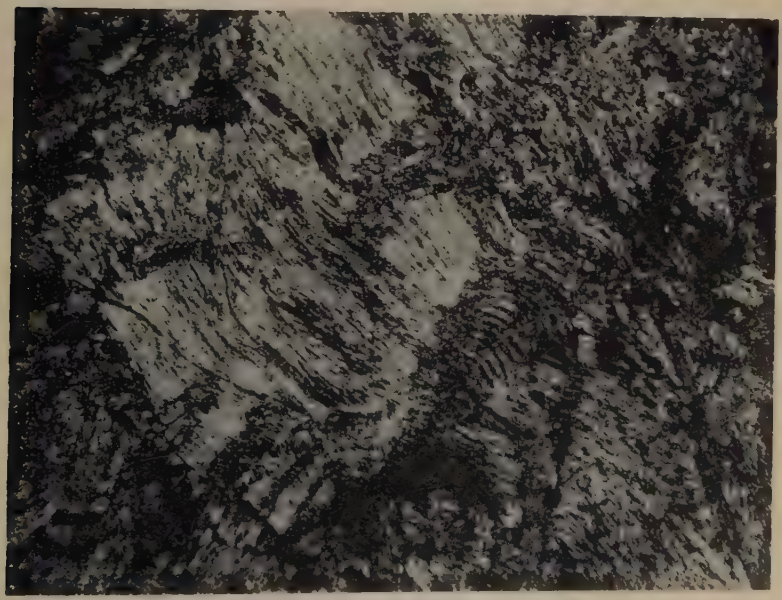

Fig. 6.

Figs. 5 and 6. (×90) Photomicrographs of pyrochroite ore (Kimiman-kô) from the Misago ore body, Noda-Tamagawa. The ore consists mainly of fibrous aggregates of pyrochroite. Fig. 5. Polarizer only. Fig. 6. Crossed nicols.

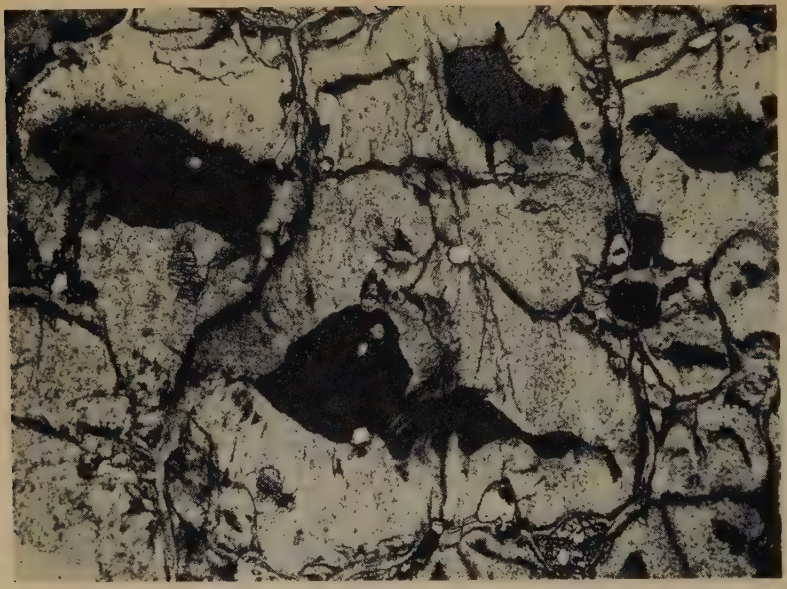

Fig. 7.

Fig. 8.

Figs. 7 and 8. (×39) Photomicrographs of manganosite-bearing pyrochroite ore (Kimiman-kô) from the Maida ore body, Noda-Tamagawa. The manganosite (dark grains) is replaced by fibrous pyrochroite owing to hydration. Fig. 7. Polarizer only. Fig. 8. Crossed nicols.

observed on the ores coincides with major folding of the Misago ore body. It is interesting to note that this folded part of the Misago ore body represents deformation zone of this mining area and is broken or cut by some faults with small displacement. Along the major faults called Misago-fault and "Penwithite"-fault, hydrothermal alteration of the ore body and country rocks is prominently developed.

The hydrothermal neotocite or penwithite occurs very commonly in fissures along the faults. Sulphide minerals such as alabandite, arsenopyrite, sphalerite, galena and molybdenite are also found in or near the fissures.

Physical and optical properties of pyrochroite

The freshly broken surface of the pyrochroite ore is as white as shown in Plate I, Fig. 2a, but, shortly after its surface was exposed in air its colour turns to dark brown as shown in Plate I, Fig. 2b.

Under the microscope it is revealed that pyrochroite is the chief constituent of the white pyrochroite ore with minor amount of secondary rhodochrosite. The pyrochroite is usually fibrous or scaly with subparallel growths as shown in Plate III, Figs. 5 and 6. When manganosite is present in the ore, it is usually replaced from its periphery by fibrous aggregates of pyrochroite, which are usually bent or twisted indicating their mechanical deformation caused by the volume increase due to hydration of manganosite into pyrochroite.

The refractive indices of colourless pyrochroite were measured by immersion method as given in Table 1.

The oxidized sample is pale brown to brown in colour and highly pleochroic (O>E).

The specific gravity of a small block of pyrochroite mass was measured as 3.32 by ordinary balance method. This value was a little higher than the known specific gravity for pyrochroite, be-

Table 1. Optical properties of pyrochroite and brucite.

	Pyrochroite (Noda-Tamagawa)	Pyrochroite (Gonzen)[9]	Pyrochroite (San Jose)[12]	Brucite (Suan)[16]
ω	1.725	1.723	1.733	1.566
ε	1.683	1.681	n.d.	1.584
$\varepsilon \sim \omega$	0.042	0.042	0.04	0.018
Optical character	−	−	−	+
Character of zone	+	+	+	−
Extinction	straight, //el to fiber	straight	straight	straight, //el to fiber
Pleochroism (oxidized sample)				
O	brown	brown	deep yellowish red to colourless	−
E	lighter brown colourless	lighter brown		−
Absorption	O>E	O>E		

cause the sample measured was not free from heavier minerals such as tephroite, rhodochrosite and alabandite.

Chemical composition

The analysed specimens of the pyrochroite ore were collected in the working place of the Misago ore body on the lower 3rd level of the Noda-Tamagawa mine. As soon as the samples were broken off from the face of the stope, they were immersed in fused paraffin in order to cover them with paraffin film. The paraffin coated samples were kept perfectly unoxidized in the core untill they were sent to the laboratory. Then, the unoxidized parts were carefully picked out and analyzed by the third author, J. Ito. The result of the analysis is given in Table 2, No. 1.

As small amounts of rhodochrosite, tephroite, galaxite and alabandite were contained in the analyzed samples, $(Mn, Mg, Ca)_2SiO_4$, $(Mn, Mg, Ca)\,CO_3$, $(Mn, Mg, Ca)\,Al_2O_4$ and MnS were reduced as impurities from the result of the analysis. The recalculated molecular

Table 2. Chemical analysis of the pyrochroite mass.

	1	1a	1b	2	3
MnO	72.92%	1.0280	0.9201...1.00	77.3%	79.74%
MnO_2	none				
$H_2O(+)$	17.42	0.9669	0.9669...1.05	20.9	20.26
SiO_2	3.13	0.0521			
TiO_2	none				
Al_2O_3	0.18	0.0018			
Fe_2O_3	0.42	0.0026			
FeO	n.d.			0.4	
MgO	1.06	0.0263		1.7	
CaO	2.01	0.0358		tr.	
CO_2	2.70	0.0614			
S	0.28	0.0087			
Alkalies	n.d.				
Total	100.12%			100.3%	100.00%
	−0.14 − O(less for S)				
	99.98				
Sp. Gr.	3.32_6			3.24	3.25

1. Pyrochroite mass containing small amounts of tephroite, galaxite, Mn-carbonate, alabandite, etc. J. Ito, analyst.
1a. Molecular proportion of 1.
1b. Less $(Mn, Mg, Ca)_2 SiO_4$ (tephroite), $(Mn, Mg, Ca)(Al, Fe)_2 O_4$ (galaxite), $(Mn, Mg, Ca) CO_3$ (Mn-carbonate) and MnS (alabandite).
2. Pyrochroite from Långban (after Sjögren)[14].
3. Calculated composition of $Mn(OH)_2$.

ratio is $[MnO]:[H_2O]=1:1.05$ in good agreement with the formula of $Mn[OH]_2$.

X-ray investigation

The X-ray powder diagrams of the pyrochroite ore (Table 3) and manganosite (Table 4) were made by means of Philips Norelco diffractometer with Fe radiation. The diagram of the pyrochroite ore indicates the presence of Mn-carbonate in the analyzed material. The X-ray powder data for the pyrochroite ore are compared with

those of synthetic pyrochroite obtained by Klingsberg and Roy[10] in Table 3. The lattice dimensions of the pyrochroite from Noda-Tamagawa were calculated as follows:

$$a_0 = 3.323\ \text{Å.}, \qquad c_0 = 4.738\ \text{Å.}$$

The X-ray powder data for the manganosite from Noda-Tamagawa

Table 3. X-ray powder data for pyrochroite ore from the Noda-Tamagawa mine, Iwaté prefecture and those of synthetic pyrochroite.

1. (synthetic)		2. (Noda-Tamagawa)					
d(Å)	I	d(Å)	I	Q_{obs}	Q_{cal}	hkl*	hkl**
4.726	100	4.743	100	0.0445	0.0446	001	
2.870	18	2.879	10	0.1208	0.1208	100	
		2.847	10	0.1234	0.1231		104
2.453	40	2.460	40	0.1652	0.1654	101	
		2.394	1	0.1745	0.1745		110
2.361	6					002	
		2.179	2	0.2106	0.2110		113
		2.005	1	0.2488	0.2489		202
1.825	26	1.829	20	0.2989	0.2990	102	
		1.774	2	0.3178	0.3178		018
1.658	6	1.661	10	0.3625	0.3623	110	
1.567	4	1.568	5	0.4067	0.4069	111	
1.381	8	1.384	5	0.5221	0.5218	103	
1.346	3					112	
		1.230	5b	0.6610	0.6613	202	
1.180	3					004	
		b=broad					
		a_0=3.323Å. (calculated mean value)					
		c_0=4.738Å. („)					

hkl* =indices for pyrochroite.

hkl**=indices for rhodochrosite (calculated as a_0=4.788Å., c_0=15.70Å.).

1. Pyrochroite, synthetic material. (After Klingsberg and Roy[10]).
2. Pyrochroite ore, Noda-Tamagawa mine, Iwaté prefecture. Unfiltered Fe radiation (=1.93597Å), slit: 1°-0.006″-1°, scanning speed: 2°/1 min., scale factor: 4, multiplier: 1, time constant: 4 sec.

Table 4. X-ray powder data for manganosite from the Noda-Tamagawa mine, Iwaté prefecture and those of synthetic material.

1. (synthetic)		2. (Noda-Tamagawa)				
d(Å)	I	d(Å)	I	Q_{obs}	Q_{cal}	hkl
2.568	62	2.565	30	0.1520	0.1522	111
2.223	100	2.220	100	0.2029	0.2029	200
1.571	58	1.571	30	0.4052	0.4058	220
1.340	21	1.339	10	0.5578	0.5579	311
1.283	13	1.283	10	0.6075	0.6086	222
1.1112	11	1.110	2	0.8117	0.8115	400
1.0198	10	1.019	1	0.9631	0.9637	331
0.9938	18	0.9927	10	1.0148	1.0144	420
a_0=4.445Å.		a_0=4.440Å. (calculated mean value)				

1. Manganosite, synthetic material (After Swanson, Gilfrich and Ugrinic[15]).
2. Manganosite, Noda-Tamagawa mine, Iwaté prefecture. Unfiltered Fe radiation (=1.93597Å.), slit: 1°-0.006″-1°; 4°-0.006″-4°, scanning speed: 2°/1 min., scale factor: 4, multiplier: 1, time constant: 4 sec.

are compared with those of synthetic material obtained by Swanson et al.[15] in Table 4.

Origin

Pyrochroite has hitherto been known as a hydrothermal vein mineral and its occurrences were recorded only in a few localities. It has not been recorded that pyrochroite occurs as fibrous aggregates constituting important manganese ores as in the case of the Noda-Tamagawa mine where, as described in the previous sections, relict grains of manganosite are found in the massive aggregates of pyrochroite. The textural relation of the pyrochroite [$Mn(OH)_2$] to manganosite [MnO] resembles very much that of brucite [$Mg(OH)_2$] to periclase [MgO] in pencatite or predazzite as shown in Plate II, Figs. 3 and 4.

Periclase-bearing brucite-marbles, which are often called pen-

catite or predazzite, occur in inner contact aureoles of granitic masses and are interpreted as dedolomitization product through the following two stages;

$$\underset{\text{(dolomite)}}{CaMg(CO_3)_2} = \underset{\text{(calcite)}}{CaCO_3} + MgO + CO_2$$

$$\underset{\text{(periclace)}}{MgO} + H_2O = \underset{\text{(brucite)}}{Mg(OH)_2}$$

Judging from similar textural relation the pyrochroite ore may be interpreted as hydration product from manganosite ore. It is highly probable that the original manganese ore composing the Misago ore body was a bedded rhodochrosite deposits of sedimentary exhalative type as mentioned in the previous paper[17]. During the period of the contact metamorphism, thermal dissociation of rhodochrosite ore may have taken place and resulted in the formation of manganosite. Manganosite is by no means a rare mineral in the Noda-Tamagawa mine.

In the folded and faulted zones where openings or channels are provided for fluids, thermal dissociation of rhodochrosite will take place at lower pressure and temperature than in undisturbed places. Through the openings, through which CO_2 gas escaped at the time of dissociation of rhodochrosite, the water emanated from magma will have access in the later stage of magmatic activity. Therefore, the manganosite ore once formed, may have been altered to pyrochroite ore in the later stage. The extensive development of the unique pyrochroite ore in the Misago ore body is probably due to such structural control.

In order to discuss the genesis of the pyrochroite ore, the relative position of P-T curves for univariant equilibrium of some reactions relating to thermal metamorphism of manganese carbonate deposits is shown in Fig. 9. The known parageneses of minerals constituting the hornfelses and skarns found in the Noda-Tamagawa mining area indicate that the grade of contact metamorphosed rocks belongs to the hornblende or pyroxene hornfels facies. As shown in Fig. 9 the dissociation curves (e or g) of manganese carbonate (rhodo-

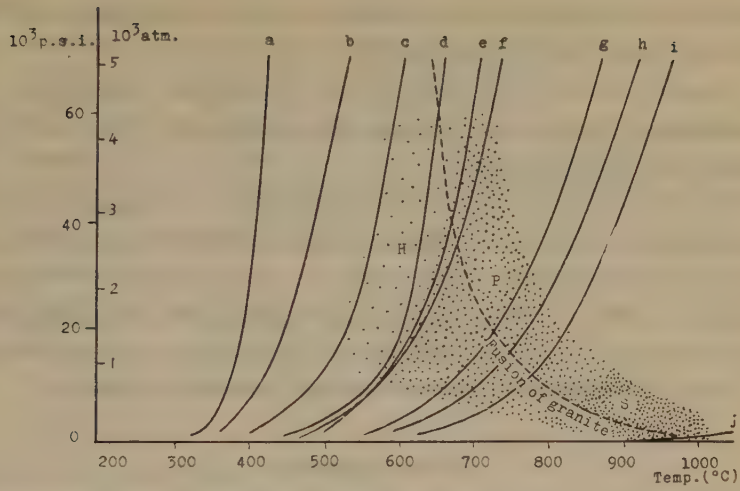

Fig. 9. Relative position of P (fluid)-T curves of univariant equilibrium for reactions relating to the thermal metamorphism of Mn-carbonate, magnesite, limestone and dolomite.

a) $Mn(OH)_2 \rightleftarrows MnO + H_2O$ (experimentally determined by Klingsberg and Roy[10]) (pyrochroite) (manganosite)

b) $2MnCO_3 + SiO_2 \rightleftarrows Mn_2SiO_4 + CO_2$ (theoretically calculated by Yoshinaga[20]) (rhodochrosite) (quartz) (tephroite)

c) $MnCO_3 + SiO_2 \rightleftarrows MnSiO_3 + CO_2$ (calculated by Yoshinaga[20]) (rhodochrosite) (quartz) (rhodonite)

d) $Mg(OH)_2 \rightleftarrows MgO + H_2O$ (experimentally determined by Kennedy[9]) (brucite) (periclase)

e) $MnCO_3 \rightleftarrows MnO + CO_2$ (calculated by Yoshinaga[20]) (rhodochrosite) (manganosite)

f) $CaCO_3 + SiO_2 \rightleftarrows CaSiO_3 + CO_2$ (calculated by Danielson[2]) (calcite) (quartz)

g) $MnCO_3 \rightleftarrows MnO + CO_2$ (experimentally determined by Goldsmith and Graf[6]) (rhodochrosite) (manganosite)

h) $MgCO_3 \rightleftarrows MgO + CO_2$ (experimentally determined by Harker and Tuttle[8]) (magnesite) (periclase)

i) $CaMg(CO_3)_2 \rightleftarrows CaCO_3 + MgO + CO_2$ (Harker and Tuttle[8]) (dolomite) (calcite) (periclase)

j) $CaCO_3 \rightleftarrows CaO + CO_2$ (Weeks[18][19]) (calcite) (lime)

H: Field of the hornblende-hornfels facies.

P: Field of the pyroxene-hornfels facies.

S: Field of the sanidinite facies. (After F. J. Turner)

chrosite) lie between the dissociation curve of dolomite and the curve of formation of wollastonite.

Thus it is possibly considered that the partial dissociation of rhodochrosite took place at some favourable places in the Noda-Tamagawa contact aureole.

The P-T curve for the reaction pyrochroite=manganosite+H_2O is denoted by a in Fig. 9. Manganosite is unstable in the fields of lower temperature side of the curve a. It is also interesting to note that neotocite, bementite, secondary manganese-carbonate, manganese-bearing clay minerals and various sulphides have been formed in connection with the above mentioned pyrochroitization.

Acknowledgements

The authors wish to express their sincere thanks to Mr. K. Saida, Superintendent of Noda-Tamagawa Mine and Messrs. A. Sato and K. Fukuda, mining geologists of the mine, and Mr. S. Yui, graduate student of the University of Tokyo for their kind assistance during field works. Special thanks are also due to Prof. N. Katayama and Assist. Prof. M. Minato for their advice and help on the occasion of taking the photograph of pyrochroite. Dr. S. Takasu kindly aided their X-ray works. The authors are also indebted to the Ministry of Education for a Grant from the Government Expenditure for Scientific Research.

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Manuscript received January 30, 1960.

Mineralogical Journal, Vol. 3, No. 1, pp. 42-44, Feb., 1960

SHORT COMMUNICATIONS

PYROMORPHITE FROM THE YAWA TIN LODE MINE, BURMA

Toshio Kato

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Introduction. The pyromorphite samples from the Yawa Tin Lode Mine, which were collected by Mr. Kamezo Matsuda of the Sumitomo Metal Mining Co. during his stay in Burma in 1955, were supplied to the writer for study through the courtesy of Prof. Nobuo Katayama of University of Tokyo. The present paper describes its mineralogical data.

*Locality and occurrence**. The mine lies forty-four kilometres east of Palauk between Tavoy and Mergui in the southeastern part of Burma. The region is composed of Mergui series of older Palaeozoic system which mainly consists of sandstone and phyllitic slate. The mine is working cassiterite ore which occurs in quartz veins filling the fissures in these sedimentary rocks. The principal minerals associated are pyrite, chalcopyrite and arsenopyrite. The pyromorphite was found in druses in the oxidized zone of the ore veins accompanied by limonite.

General character and optical properties. The mineral occurs as aggregates of stout hexagonal prism greenish to dark brownish yellow in colour, adhering on the surface of the country rock.

Most of the crystals display short prismatic form compose of $m(10\bar{1}0)$ and $c(0001)$ with rare small faces of $x(10\bar{1}1)$. Occasionally

* Personal communication from Mr. K. Matsuda.

m faces are curved and striated parallel to the zone [0$\bar{1}$10]. The greenish yellow to yellow coloured crystals give better reflexion than the dark brownish coloured. Parallel growth is common and twinning is rarely observed. Radioactivity was not detected. Pleochroism: O pale yellow and E nearly colourless. Absorption: O>E. Optically uniaxial negative.

Chemical composition. Spectrographic analyses were carried out on the two varieties, greenish yellow to yellow and brownish yellow. Both of them gave the similar results as follow:

Major elements: Pb, P.
Minor elements: Ag, As, Bi, Ca, Cr, Cu, Fe, Si.
Trace elements: Al, Cd, Mg, Mn, Sn, Zn.

In, K, Ni, Ti, V and W were looked for but were not found.

The greenish yellow to yellow coloured specimen was analysed chemically, with the following result:

PbO 80.52%, P_2O_5 16.18%, As_2O_5 0.90%, Cl 2.40%, $-O(=Cl)$ 0.56%, total 99.44%. (Analysed by T. Kato (1957))

A small amount of arsenic replaces phosphorus, while calcium and vanadium are nearly absent.

The relative isotopic abundance of lead determined by the ionization method follows:

$Pb^{204} : Pb^{206} : Pb^{207} : Pb^{208} = 1.0 : 17.4 : 14.7 : 36.9,$

or $Pb^{206} : Pb^{207} : Pb^{208} = 25.2 : 21.3 : 53.5.$

It is in the range of ordinary lead after Cannon et al.[1], and of relatively young age.

X-ray powder diffraction patterns. X-ray powder diffraction patterns for the same specimen were obtained by 'Norelco' X-ray diffractometer (Table 1). Their features are in agreement with those of pure pyromorphite[2]. The diffraction lines in the higher

1) Cannon, R. S., L. R. Stieff and T. W. Stern: Proc. 2nd U. N. Internat. Conf. Peaceful Uses A. E., Vol. 2, 215 (1958).

2) Ishimori, T., H. Waki and K. Muta: Jour. Min. Soc. Japan, 2, 172 (1955) (in Japanese).

Table 1. X-ray powder diffraction patterns of pyromorphite from the Yawa Tin Lode Mine, Burma. (Cu$K\alpha$ radiation)

hkil	*d*	*I*	*hkil*	*d*	*I*
$11\bar{2}0$	4.9 Å	10	$50\bar{5}0, 30\bar{3}3$	1.722 Å	5
$20\bar{2}0$	4.4	25	$33\bar{6}0$	1.682	5
$11\bar{2}1$	4.2	40	$42\bar{6}0$	1.632	10
0002	3.67	10	$33\bar{6}1$	1.627	5
$10\bar{1}2$	3.38	30		1.601	8
$21\bar{3}0$	3.28	35	$51\bar{6}0$	1.548	20
$21\bar{3}1$	2.990	80	$33\bar{6}2$	1.526	20
$11\bar{2}2$	2.957	100	$33\bar{6}3$	1.364	5
$30\bar{3}0$	2.900	60	$60\bar{6}2$	1.349	5
$12\bar{3}2$	2.440	5*b*	$61\bar{7}0$	1.318	7
$13\bar{4}1$	2.266	15		1.301	6
	2.199	20	$44\bar{8}0$	1.252	6
$40\bar{4}0$	2.164	10	0006	1.222	3
$22\bar{4}0$	2.065	70		1.208	3
$31\bar{4}2$	2.004	20		1.191	4
$21\bar{3}3$	1.959	30		1.173	5
	1.865	25		1.149	2
$41\bar{5}0, 0004$	1.833	10			

35kV-15mA, 4-1-4. 1°/min.

angle are rather broad and show compositional variation in narrow range. The lattice constants determined are $a_0=10.00$Å and $c_0=7.33$Å. The X-ray study on the dark brown specimen gave the same results.

Acknowledgement. The writer wishes to express his thanks to Prof. N. Katayama for his courtesy, to Mr. K. Matsuda of the Sumitomo Metal Mining Co. for supplying this valuable specimen, and to Mr. J. Okamoto of Hitachi Central Research Institute for his guidance in the measurement of the isotope ratio.

ABSTRACT OF THE KOBUTSUGAKU ZASSHI,

JOURNAL OF THE MINERALOGICAL SOCIETY OF JAPAN, VOL. 4, NO. 3,

June, 1959

On Trevorite (pp. 137-147)

Shin-ichi Takasu

Regarding spinel type compounds, the writer established a method of determining the distribution of cations between A and B sites. The method is based on the observed values of $I_{hkl\lambda_1}/I_{hkl\lambda_2}$, where λ_1 and λ_2 are the wave lengths of X-rays and are selected so as to give rise to anomalous dispersion due to the cation in question. As an application of the method, the x value of an artificial trevorite $Fe_xNi_{1-x}(Fe_{2-x}Ni_x)O_4$ was determined by the use of Fe $K\alpha$ and Fe $K\beta$. The trevorite was prepared by sintering method and it was revealed that the values of x, T°K. (sintering temperature) and μ_B (saturation magnetization) of the trevorite satisfy the formulae derived on the basis of Boltzman's statistical mechanics.

On Acicular Calcite from the Vicinity of the Ezo-Banryu Limestone Cave, Tôma-mura, Hokkaido (pp. 148-150)

Zyunpéi Harada and Masaru Gotô

Acicular calcite occurs as fibrous aggregate in cavities of limestone at the vicinity of the Ezo-Banryu limestone cave, central Hokkaido.

Under the microscope, the individual crystals of the acicular calcite are transparent, and of very fine needle shape up to 1 mm. in length and to 50μ. in width. Their maximum extinction angle with the crystal elongation is about 48°. X-ray and chemical investigations show that they are almost pure calcite, although traces of magnesium are indicated by the spectrographic analysis.

A Regularly Interstratified Mg-Montmorillonite-Chlorite from the Tsunemi Dolomite Deposit, Fukuoka Pref. (pp. 151-156)

Hiroshi Takahashi

A clay, which was found filling small fissures in the dolomite deposit of Tsunemi, has been studied under the microscope and the electron microscope, and by X-ray powder method and chemical analysis. From the results the writer has drawn the conclusion that the clay is composed of a regularly interstratified Mg-montmorillonite-chlorite which is rather rich in magnesia but poor in alumina and has the spacing of 29.5 Å.

On the Single Crystals of Jacobsite from San-no-také, Fukuoka Pref., and their Oxidation by Heating (pp. 157-176)

Goro Shibuya

A mineral from San-no-také, which was once described as magnetite with crystal faces of *d*(110), *m*(311) and *o*(111), has been revealed to be jacobsite. It occurs as single crystals up to 3 cm. across, with chemical composition of $(Mn^{2+}_{0.38}\ Fe^{2+}_{0.60})\ Fe^{3+}_{2.01}\ O_{4.00}$, and with unit cell parameters of $a_0 = 8.4370 \pm 0.0005$ Å. Its Curie point is at $460° \pm 3°$C. and saturation magnetization is 125 e.m.u./g. The reflected light from the polished section is pale olive green.

When its powder is heated in air, it is oxidized into black, ferromagnetic material. The d. t. a. curve displays exothermic peaks at 290°, 540° and 800°C. The oxidation may be performed in two steps. First, Fe^{2+} at B site is oxidized into Fe^{3+} and vacancy is introduced at B site at about 290°C. Second, the vacancy disappears and maghemite phase separates from jacobsite and subsequently oxidized into hematite at about 540°C. The resultant oxidation product is the mixture of jacobsite with about 30 mol.% of Mn_3O_4 and the (Mn-)hematite, accompanied by a small amount of Mn_3O_4 (?).

Cesium Content of Beryl (pp. 177-183)

Shunji Matsuda

Cesium content of beryl has been determined on eight specimens from Japan, six from Korea and one from Mongolia. Determinations were made

with a flame photometric technique refined by the author, in the presence of potassium (500 p.p.m.) after the elimination of Si, Al and Be by wet method. The range of the cesium concentration in those specimens was from 10^{-3}% to 10^{-2}%. The result indicates the absence of correlation between the concentration of cesium and that of potassium.

Ferberite from the Ashio Copper Mine, Japan (pp. 184-191)

Takeshi Nakamura

Ferberite from the Ashio copper mine, Tochigi Prefecture, with a composition near the Fe end member of wolframite series has been described. The mineral occurs as tabular, or slender needle crystals in cassiterite-bearing quartz veins in rhyolite. Chemical analysis gave the following percentages and formula: WO_3 75.95, FeO 22.49, MnO 0.98, CaO 0.08, MgO 0.04, total 99.54%; $(Fe_{0.96}\ Mn_{0.04})\ WO_4$. Using the X-ray diffractometer, lattice constants, $a_0=4.721$ Å., $b_0=5.710$ Å., $c_0=4.951$ Å. and $\beta=90°00'$, were obtained. Calculated density is 7.556 g/cm^3.

It is inferred that slender needle or thin platy ferberite associated with colloform variety of cassiterite, "wood tin", is a characteristic mineral of the Cenozoic "subvolcanic" or "xenothermal" deposits in Japan. Such occurrences have been found from the Ikuno and Akenobé mines, Hyogo prefecture, the Ashio mine, Tochigi prefecture, etc.

Moreover, huebnerite, with a composition near the Mn end member of wolframite series has also been found from the Cenozoic "subvolcanic" deposits of Ikuno, and also from the Nishizawa mine, Tochigi prefecture. While, wolframites of intermediate compositions have been reported from pegmatitic or hypothermal deposits in Japan. The modes of occurrence and mineralogical properties of wolframite series are very interesting from the view point of ore genesis.

Short Communications

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Mumeikai Group

40. *Descloizite from the Miyoshino Mine, Fukuoka Pref.*

Yohachiro Okamoto, Kin-ichi Sakurai and Akira Kato

A yellowish grey crust found in the gold-quartz vein of the Miyoshino mine has been determined as descloizite on the basis of X-ray powder patterns and of chemical analysis.

41. *Descloizite from the Yasaka mine, Yamaguchi Pref.*

Hideo Inoué, Kin-ichi Sakurai and Akira Kato

Descloizite occurs filling small fissures in a vein and the country rocks at the Yasaka mine. It gives X-ray powder patterns similar to the specimen from the Miyoshino mine.

Reviews

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Printed by "GAKUJUTSU" Printing Co., Ltd.

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PUBLICATIONS

Kôbutsugaku Zasshi, Journal of the Mineralogical Society of Japan

A journal containing articles and reviews on mineralogy and allied sciences contributed by members of the Mineralogical Society of Japan. Published every four months, in Japanese. The price of subscription is $1.00 per single copy.

Mineralogical Journal

A journal containing articles on mineralogy and allied sciences, published semi-annually in English principally. Contributions are invited from every-one. The Journal completed its second volume of six numbers in 1959. Back numbers may be obtained at $1.00 per single copy for the time being. The price of subscription to third volume is $5.00 per volume.